

Dynamic Characteristics of Drop-substrate Interactions in Direct Ceramic Ink-jet Printing using High Speed Imaging System

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ABSTRACT

Solid freeform fabrication has the potential to construct ceramic parts, directly from computer aided design (CAD) data, without a mould or a die by the addition of material. Direct ceramic ink-jet printing is one of the techniques used in freeform fabrication. Ceramic tiles used in space vehicles can be produced by this method wherein a porous ceramic substrate (Al_2O_3/SiC) can be filled with a ceramic ink and processed subsequently. The success of this process depends on the systematic preparation of ceramic inks and the deposition of the ceramic ink on the substrate. In this paper, photographic studies were made on the characteristics of ceramic ink droplets when these are deposited on a porous ceramic substrate from a burette under gravity. For this investigation, ceramic inks were prepared using different amounts (0.25–3.0 vol. %) of an organic dispersant (oleic acid) added to a ceramic composition containing different amounts: (a) (7.5–17.5 vol. %) of alumina and (b) (7.5–15.0 vol. %) of zirconia with ethyl alcohol as a carrier. From this study, the drop formation, sedimentation in the drop, spread of drop on the substrate, splashing of drop impinging a previous ceramic ink layer on the substrate, and merging of droplets after deposition, are observed. This method is useful for manufacturing of parts with ceramic fibres filled with ceramic particles and this study can provide inner details on the behaviour of ink drops.

Keywords: Direct ceramic ink jet printing, solid freeform fabrication, drop formation, drop impact, splashing, drop spread, ceramic particles sedimentation, ceramic substrate

1. INTRODUCTION

Solid freeform fabrication is a potential process to construct parts without the use of conventional tooling directly from computer-aided design systems which allows the forming of complex shapes¹. The techniques under freeform fabrication are: Stereolithography², selective laser sintering³, 3-D printing⁴, fused deposition of ceramics⁵, laminated object manufacturing, and direct ceramic ink-jet printing¹(DCIJP).

The DCIJP finds applications in the fabrication of complex-shaped monolithic ceramic parts, ceramic composites, solid-oxide fuel cells, integrated circuits, components of fuel cells, gas turbines, ceramic tiles used in space vehicles, and many other equipments operating at high temperature owing to their ability to retain strength at high temperature. Ceramic pottery industry can also benefit from this process.

The DCIJP uses ceramic powder presented in a carrier medium which is deposited using a delivery system actuated by a piezoelectric device. A mathematical model for droplet formation from orifice and its deposition on substrate in DCIJP has been developed by Vijay and Prakasan⁶. The success of this process depends on the systematic preparation of ceramic inks, droplet formation in the print head, and droplet deposition on the substrate. It can be used to fabricate larger parts like ceramic tiles for aerospace vehicles

which are used as heat shields for the critical units of the space vehicles.

2. PRESENT WORK

An extensive literature survey was carried out on the various methods deployed by the researchers on DCIJP. It was understood that there is enough scope to carry out research in the area of depositing the ceramic ink on the ceramic substrate which can be applied for the faster and less expensive fabrication of complex monolithic ceramic parts. These results can provide the researchers with valuable information on the characteristics of deposition of ceramic ink droplets on ceramic substrate. When a large scale manufacture of ceramic components is attempted, the characterisation of drops, their spread, and splashing are importance to the process optimisation. With such an objective this work was taken up.

3. OBJECTIVES

First, a set of experiments was conducted on DCIJP. The research work studied the optimum composition for preparing ceramic ink, developed a database on rheological properties of the ceramic inks, analytically determined the packing density for the good flow characteristics, and simulated flow through print head. Objectives of the present

work were to:

- Study the different phases of drop formation and stretching of ink droplet while deposited from a burette using high speed imaging system,
- Understand the behaviour of the drop while impinging the ceramic substrate and its spread when the ink is deposited for first time at different stand-off distances and ink compositions, with a high speed camera. (this was repeated for drop on drop and multiple depositions), and
- Study the convergence of two drops while deposited at different stand-off distances and ink compositions-on-the-substrate.

4. EXPERIMENTAL DETAILS

The experimental details while depositing ceramic inks on a ceramic substrate have been discussed and the setup for droplet-deposition is presented:

4.1 Preparation of Ceramic Ink

An electronic balance from Shimadzu Corporation, Japan, with a capacity (220 g), readability (0.001g), type BL-220H, was used for accurately weighing the ceramic particles. Ceramic inks were prepared using ultrasonicator, Sonics Vibra Cell, USA. The frequency set was 20 KHz. The apparent viscosity of suspension (with different vol. per cent of ceramic and dispersant) was measured using Brookfield digital viscometer Model DV-E. The preparation of alumina and zirconia-based ceramic inks is extensively discussed by RamaKrishnan⁷. The selection of appropriate binders and dispersants combined with high shear and ultrasonic mixing to produce ceramic components by multilayer printing has been reported by Song⁸.

Zirconia (ZrO_2) having a density of 5.5 kg/m^3 , molecular weight of 123.22 and alumina having a density of 3.96 kg/m^3 and molecular weight of 101.96 were used in this work. Ethyl alcohol of 99.9 per cent purity was used as the carrier. Prevention of ceramic ink flocculation can be controlled effectively by using dispersant that introduces repulsion between ceramic particles to counteract attractive forces. The addition of 1–2 wt per cent of oligomeric dispersant corresponds to the highest adsorption level on the powder, the maximum sediment-packing efficiency, the minimum ink viscosity, and high sintered density⁹. So oleic acid was used as the dispersant.

4.2 Selection of Different Apparent Viscosities of Ceramic Inks

Ceramic inks with different compositions can be made by: (i) 7.5–17.5 vol. per cent of alumina, and (ii) 7.5–15 vol. per cent of zirconia in ethyl alcohol with different dispersant content varying between 0.75–3 vol. per cent. The optimum composition for preparing ceramic inks that can be used for DCIJP was determined and a database on rheological properties of the ceramic inks had been developed by KrishnaPrasad¹⁰ was used. From this database, different compositions were chosen: Low apparent viscosity with

the composition 7.5 vol. per cent of alumina in ethyl alcohol and 7.5 vol. per cent of zirconia in ethyl alcohol, medium apparent viscosity with the composition of 13.5 vol. per cent of alumina in ethyl alcohol and 12.5 vol. per cent of zirconia in ethyl alcohol, and high apparent viscosity with the composition of 17.5 vol. per cent of alumina in ethyl alcohol, and 15 vol. per cent of zirconia in ethyl alcohol. Dispersant of 1 vol. per cent was added in all these ceramic inks. Different apparent viscosities of inks were used for the study of droplet deposition characteristics on a substrate under gravity.

4.3 Properties of Substrate and Deposition of Ceramic Ink

A ceramic board from Murugappa Morganite Ceramic Fibres Ltd, India, with a thickness of 3 mm was used in this work. It could withstand temperature up to $1260 \text{ }^\circ\text{C}$. This has a density 240 kg/m^3 and thermal conductivity 0.113 W/mK at mean temperature. This board was used as substrate for the study of ceramic drop deposition and spread on substrate.

For different compositions of ceramic ink, impact of drop and its spread on ceramic substrate was observed by a high-speed digital imaging system from MotionPro-Redlake, USA, to understand the characteristics of ink deposition on the substrate. As DCIJP involves the flow of viscous suspension in multiple microchannels, in this study the deposition was done by dropping the ceramic ink from a burette with two nozzles under gravity (Fig. 1). This helps in investigating the spread, convergence of two droplets, and approximate size of spread on a ceramic substrate. The current setup to demonstrate the ink droplet formation and impact on hydrophobic and hydrophilic surfaces was similar to the setup used by Bhola and Chandra¹¹, Carr¹², and Roy and Jeffrey¹³.

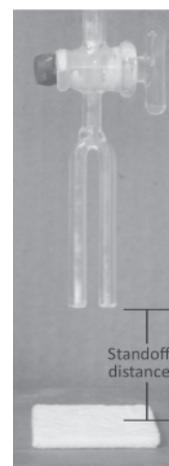


Figure 1. Experimental setup for the study of deposition of ceramic ink drop on ceramic substrate.

Figure 2 shows the facility used for ink deposition. The ceramic ink droplets were deposited from a burette under gravity. To manipulate the position for deposition

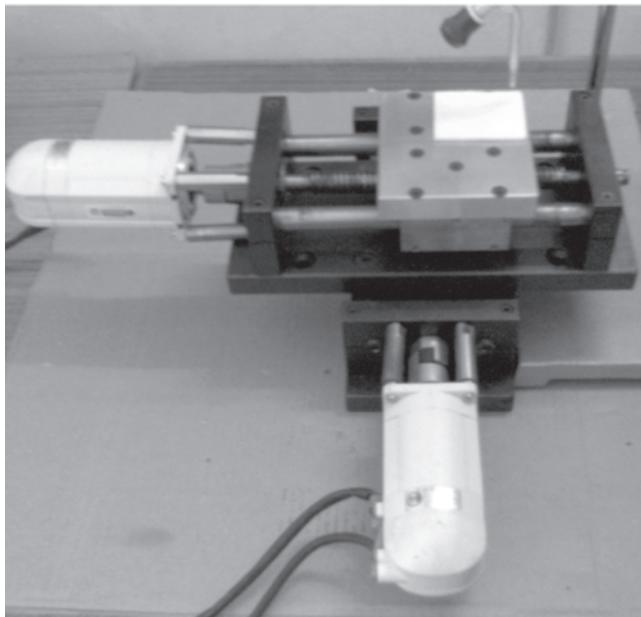


Figure 2. X-Y table for position control.

of ceramic ink droplet on a substrate, an X-Y table with 100 mm x 100 mm size was made, fixed with Panasonic servomotor and controlled by Galil motion controller PCI. Operation of this setup is used for the study of the characteristics of droplets while deposited on the substrate.

5. RESULTS AND DISCUSSIONS

5.1 Drop Formation

The dynamics of ceramic ink drop formation from burette nozzle under gravity were studied experimentally using digital imaging system with an interframe time of 0.005 s. From Fig. 3, the main stages of formation of ceramic ink drop were observed which included ejection, stretching of liquid (1-3), contraction of liquid thread (4-5), breakup of thread into the primary drop (6), and satellite formation by pinch-off of liquid thread from nozzle exit (7-10). Satellite formation occurs because of the break of liquid thread.

However, satellite formation was to be avoided in DCIJP. Satellite formation depended on three factors:

- Length of the liquid thread,
- Speed of contraction of liquid thread, and
- Time of liquid thread pinch-off.

These factors were related to the geometry of nozzle, pressure applied, viscosity, and surface tension of ceramic ink. The results of this investigation were reflected in the photographic studies by Carr¹², and Roy and Jeffrey¹³.

Applied pressure is important when the drop is produced by an actuation mechanism like piezoelectric actuation of a diaphragm above a chamber. The force exerted by the diaphragm influences drop shape. Surface tension is a major factor in controlling the drop formation and its spread. When applied pressure and surface tension were not at the optimum level, the drop breaks into smaller drops from the stretched portion as seen in the photographs. Stretching is caused by gravity, viscosity and surface tension, and the amount of liquid ejected. If these were controlled, stretching would be minimum and satellite formation could be avoided. The amount delivered will depend on the pressure and force balance is achieved by surface tension. Thus, keeping viscosity at permissible level, pressure and surface tension play an important role in the satellite formation.

5.2 Drop Impact on Substrate

Figure 4 shows the sequential images of ceramic ink drop impinging on the substrate. The drop experienced retraction and oscillation before it reached the equilibrium state. At the same time carrier medium of the ink was absorbed by the substrate and only the ceramic particles were deposited on the substrate.

Figure 5 shows that for alumina-based ink, with varying compositions there was no splashing observed for first drop impinging the substrate at stand-off distances 50 mm and 100 mm. Splashing of first droplet is observed on the surface of the substrate at stand off distances 150 mm and 250 mm for the low viscous and medium viscous alumina-

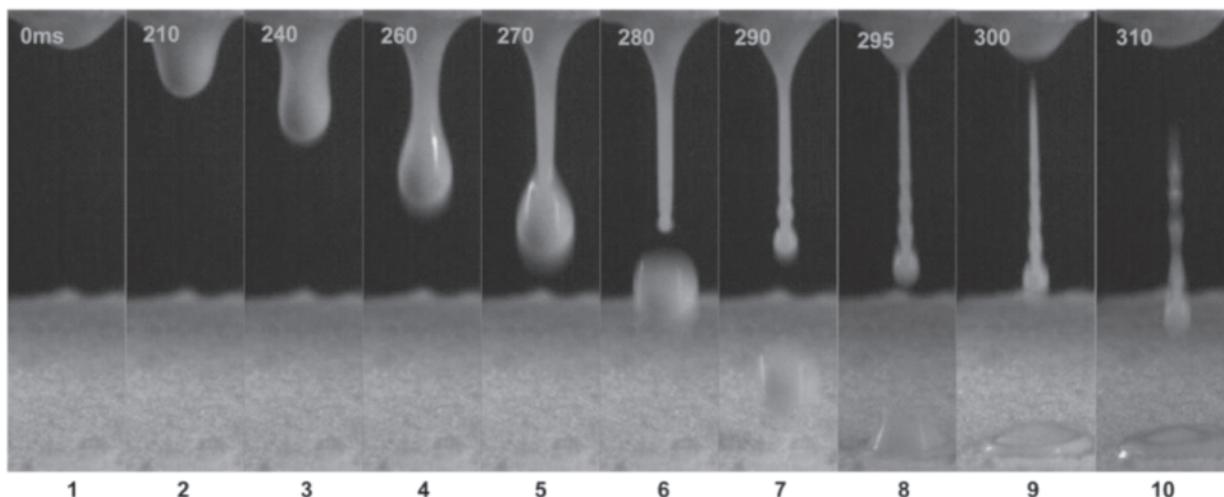


Figure 3. Sequence of formation of ceramic ink drop under gravity from burette nozzle. nozzle dia=2 mm, stand-off distance from substrate=25 mm, interframe time=0.005 s (ethyl alcohol, 13 vol % of alumina and 1 vol % of dispersant).

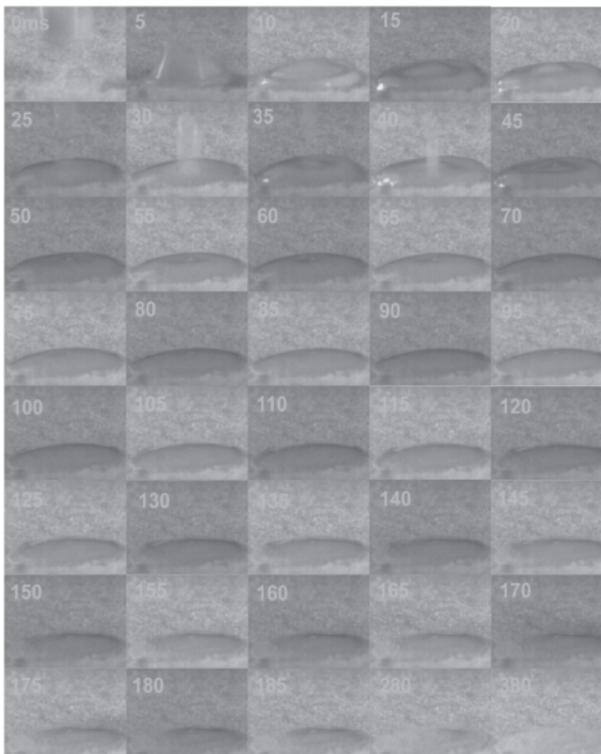


Figure 4. Sequence of ceramic ink drop impinging ceramic substrate under gravity from burette nozzle. nozzle dia=2 mm, stand-off distance from substrate=25 mm, interframe time=0.005 s (ethyl alcohol, 13 vol % of alumina and 1 vol % of dispersant).

based ceramic inks. Similar behaviour was observed for zirconia-based ceramic inks also. Carr¹⁴, investigated the fundamental mechanism of droplet formation and interaction with a substrate when applied to textile inkjet printing.

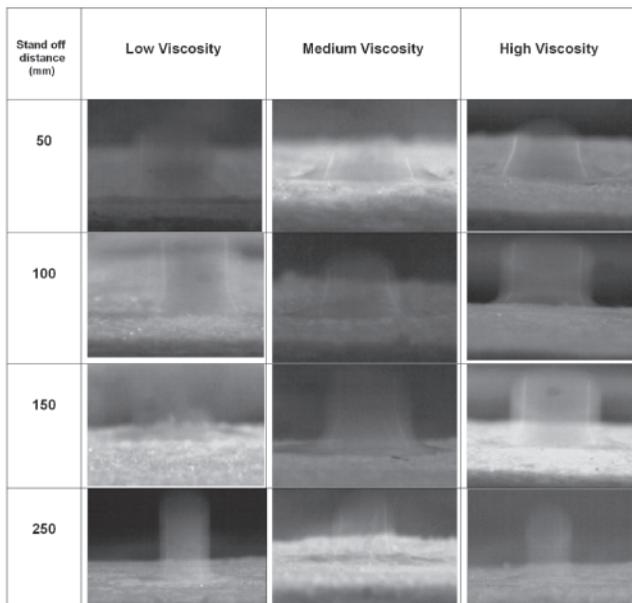


Figure 5. First drop impact on substrate under gravity, for different apparent viscosities of alumina-based ink and for different stand-off distances from substrate.

Carr¹⁵ studied the effect of solid-filled ink properties on drop formation in ink-jet systems and attempted to develop a model for predicting the effects of ink properties on drop impaction and spread on textile surfaces. Carr¹⁵ investigated the jetting behaviour in drop-on-demand ink jet systems. Carr¹⁶ studied the impaction and spreading of drops on textile substrates. These papers were the results of an ongoing research work related to textile ink-jet printing as needed by the textile industry. The current work and these papers have a similar theme applied to different products and the methodology also seems to be similar. In the present work, penetration of ink and its contribution to the improvement in properties of the substrate were important while it may not be critical in the textile applications. The investigations on the dry strength achieved in the substrate were under progress.

5.3 Drop Impact on Previous Film of Ink on Substrate

As DCIJP involves addition of material technique for developing complex large scale parts, study on the impact of ceramic ink drop on previous drop is significant. Figure 6 shows the images of drop on a film of previously deposited ceramic ink at stand-off distance 25 mm. Drops were ejected under gravity on a previous drop of ceramic ink for different stand-off distances. In Fig. 7, splashing

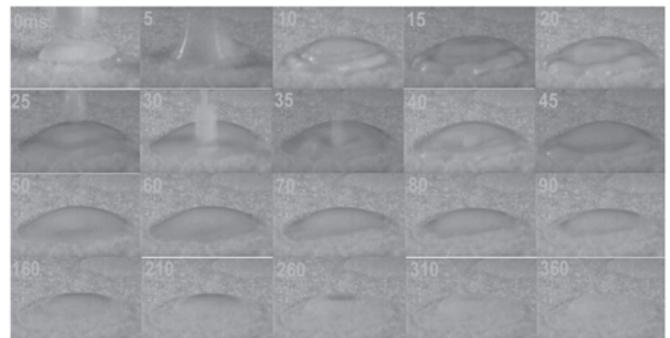


Figure 6. Sequence of ceramic ink drop impact on previous film of ceramic ink on ceramic substrate under gravity from burette nozzle. nozzle dia=2mm, stand-off distance from substrate=25mm (ethyl alcohol, 13 vol % of alumina and 1 vol % of dispersant).

was observed when second drop impacted the previous drop for stand-off distance 150 mm and 250 mm. But splashing was observed for stand-off distance 100 mm itself when third drop impacted the second drop of ceramic ink. So, for the drop deposited on previously deposited film of ink, splashing occurred as stand-off distance from the substrate increased.

Also, reduced splashing was observed for low-viscous and high-viscous ceramic inks. Figures 8(a) and 8(b) show that when low viscous ceramic inks were deposited at different stand-off distances, spread size was larger than the spread size of medium and high-viscous ceramic inks. Figures 9(a) and 9(b) show that final spreading after impact

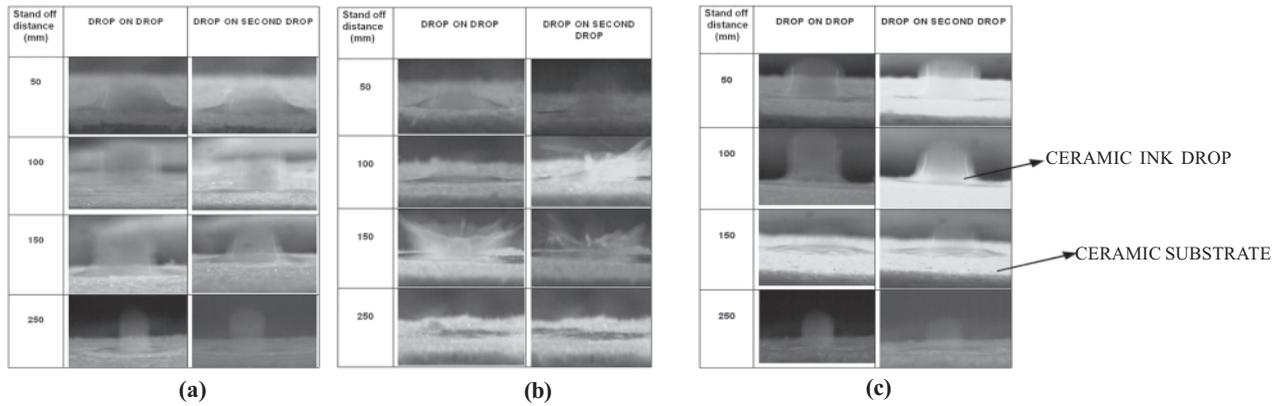


Figure 7. Drop impact on previous film of ceramic ink on substrate under gravity, for different apparent viscosities of alumina-based ink and for different stand-off distances from substrate: (a) low-viscosity ink, (b) medium-viscosity ink, and (c) high-viscosity ink.

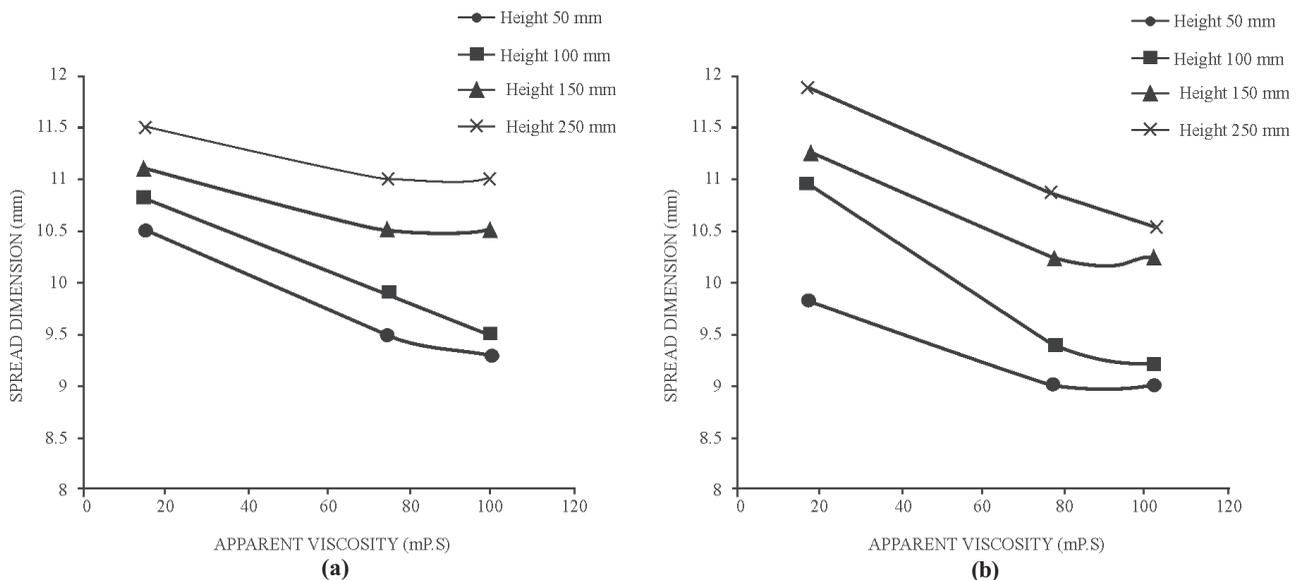


Figure 8. Variation of drop spread on substrate for different apparent viscosities of ceramic ink at different stand-off distances: (a) alumina-based ink, and (b) zirconia-based ink.

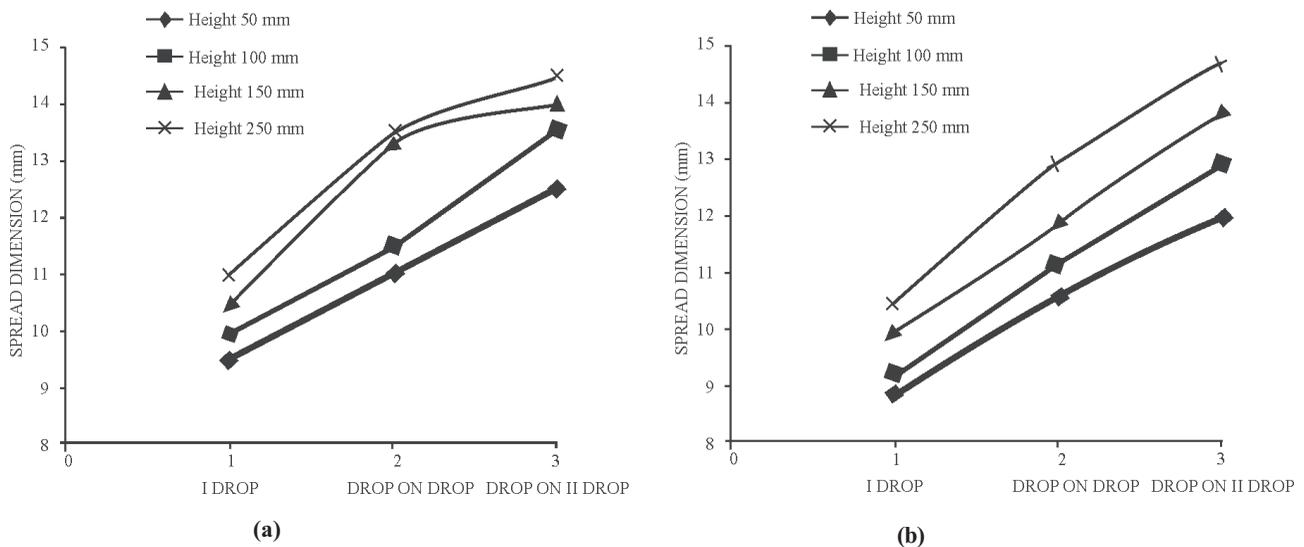


Figure 9. Variation of drop spread when drop impinges a previous drop on substrate for different stand-off distances: (a) ethyl alcohol, 13 vol % of alumina and 1 vol % of dispersant (b) ethyl alcohol, 12.5 vol % of zirconia and 1 vol % of dispersant.

was larger than for a single drop impacting the substrate. These observations can be explained as follows: The first drop settled on the substrate earlier as the carrier medium percolates leaving the particles on the substrate. But when a drop was deposited on another drop, the second drop kept its liquid content for a longer period on the previous ink layer supporting it and didn't permit the percolation of carrier into the pores easily. Thus drop on drop show better spreading nature. But drops when deposited successively didn't show an increasing spread as the edges of the deposited drops were thick and these prevented further spread and the flow started towards the centre of the drop.

5.4 Penetration of Ink Through Substrate

Qualitative studies were made on the penetration of ceramic ink through ceramic substrate for different apparent viscosities of inks. From Fig. 10, it was observed that increase in apparent viscosity of ceramic ink reduced the penetration of ink through substrate. As stand-off distance increased, penetration of ink through substrate

increased because of increase in impact velocity of ink on substrate. These measurements were made by conventional methods and the objective being the manufacture of a large component like a ceramic tile, a very high level of accuracy was not planned at stage though number of trials are carried out.

5.5 Convergence of Two Ceramic Ink Droplets and Spread

Under gravity two ceramic ink droplets were deposited from two burette nozzles. Figures 11(a), 11(b) and 11(c) show the pattern of convergence of two ceramic ink drops on substrate under gravity for varying apparent viscosities of ink and stand-off distances. Spreads were not merging for the first droplets from two nozzles. Two drops merged when drops were deposited on previously deposited ceramic ink (explanation in section 5.3). Convergence of drop depended on the distance between the nozzles, stand-off distance from substrate and velocity of droplet. When droplets were deposited, the drops could spread to the maximum extent as controlled by the substrate-droplet interface

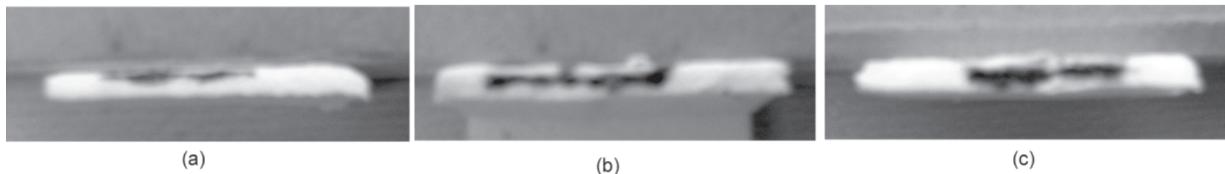


Figure 10. Cross-sectional view, penetration of ceramic ink through ceramic substrate under gravity at stand-off distance of: (a) 50mm (b) 100 m, and (c) 150 mm. (substrate thickness=3 mm, length=50 mm, and width=50 mm).

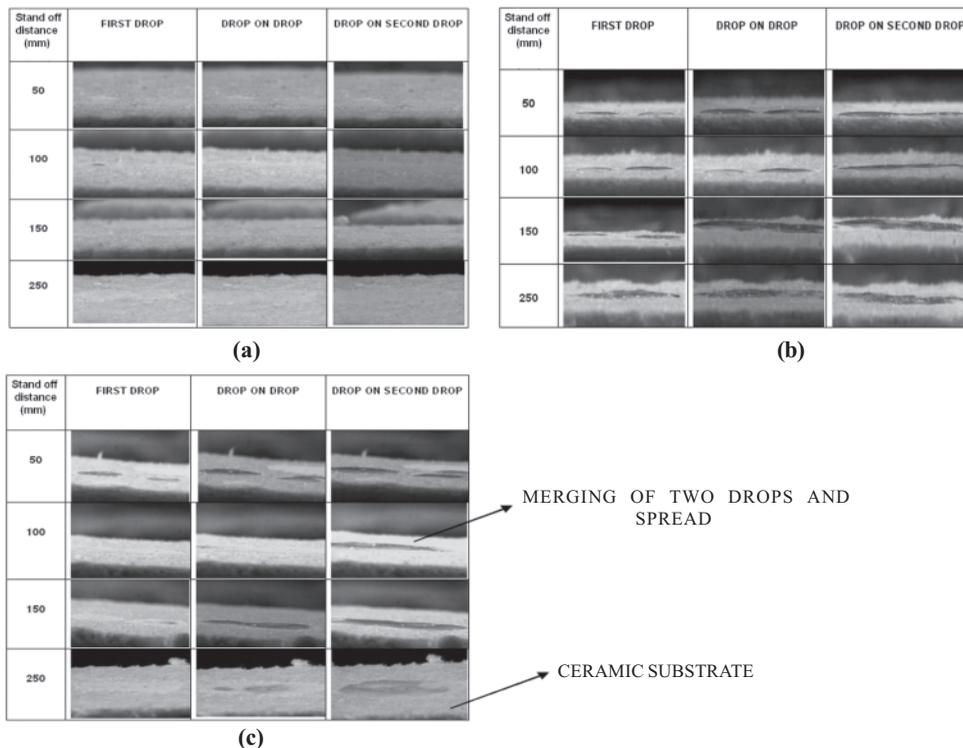


Figure 11. Convergence of two ceramic ink drops on substrate under gravity, for varying apparent viscosities of ink and stand-off distances: (a) ink with low viscosity, (b) ink with medium viscosity, and (c) ink with high viscosity.

conditions. But when the nozzles were close, the probability of merging of two drops are higher than when they were apart. Thus the difference between nozzles in the burette was a significant factor in achieving merging of drops. When the drops were deposited from a higher stand-off distance, the kinetic energy of the drop was also higher. This led to thinning of the drop (as it can flow further) when it impinged the substrate and persuaded the spread.

5.6 Behaviour of Ceramic Ink Droplet in a Manufacturing Setup

The DCIJP deposits the ceramic ink from multiple channels according to computer-defined position from CAD model. The deposition of ceramic ink droplet on substrate was done using a CNC-controlled X-Y table which moved at a speed of 5 mm/s. Figures 12(a), 12(b), and 12(c) show

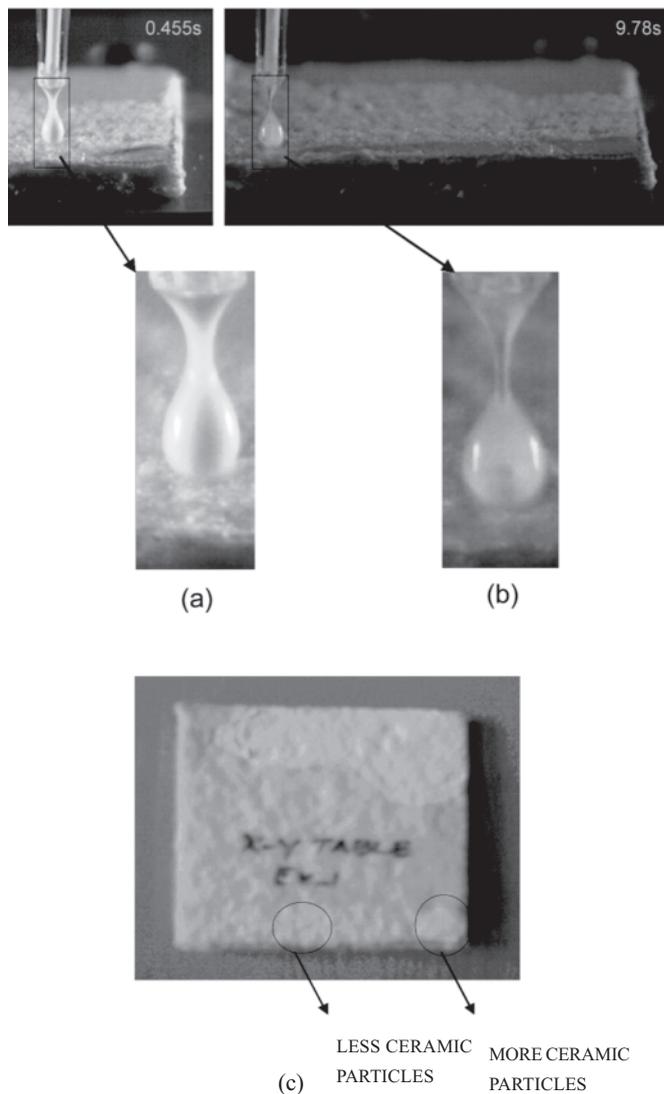


Figure 12. Settling observed in ceramic ink (ceramic ink composition: ethyl alcohol of 13 vol % of alumina and 1 vol % of dispersant). (a) ink drop with more ceramic particles, (b) ink drop with less ceramic particles, (c) ceramic ink after deposition on substrate.

that ceramic particles were more in the initial droplets but later it could be inferred that ceramic particles were less towards the end of deposition. This was noted from the appearance of drop itself as there were sedimentation taking place in the burette. As there was a time gap between the deposition of successive drops, sedimentation effects came into play and the drops in the beginning were richer in ceramic content. This pointed to an important information that the ink should be kept in such a way that sedimentation is not prominent (by adding suitable dispersants/vibration in the burette).

6. CONCLUSIONS

The present study highlights the following salient points when ceramic ink is deposited on a ceramic substrate from burette under gravity:

- Geometry of nozzle, pressure applied, viscosity, and surface tension of ceramic ink can affect the satellite drop formation. From the images of ceramic ink drop impinging on substrate, retraction and oscillation were observed before the drop reaches the equilibrium state. Also, ceramic particles settled on the surface of the substrate where carrier medium flows through the pores in the substrate. Sedimentation was also observed in the drops.
- Splashing is observed generally. Behaviour of drop on previously deposited ceramic ink was studied by the sequential images and oscillation was observed for certain time. Splashing was observed for the stand-off distance 150 mm and 250 mm when drop impacted on first layer of ink. Final spreading after impact on previous drop was larger than for a single drop impinging the substrate.
- Penetration of ink through substrate reduced as apparent viscosity of ink increased. When stand-off distance increased, penetration of ink through substrate increased.
- Convergence of two droplets was observed for various compositions and experimental conditions. This could be useful in achieving a faster deposition rate.

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